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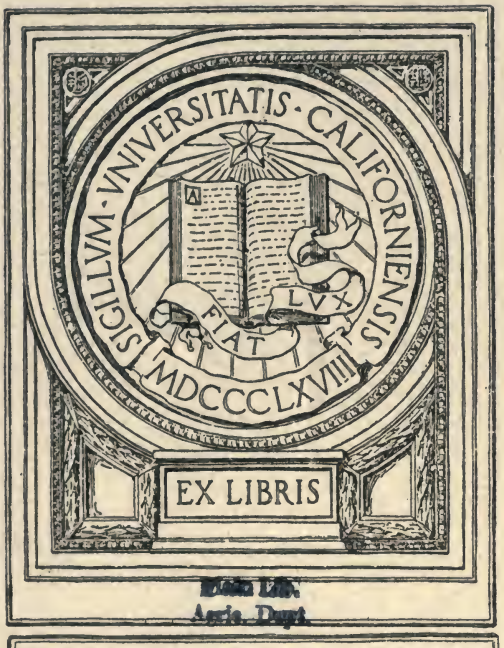

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CIRCULAR No 2.

United States Department of Agriculture,

DIVISION OF CHEMISTRY.

WASHINGTON, D. C., *December 12, 1896.*

The manuscript embracing the changes in and additions to methods of analysis adopted at the thirteenth annual meeting of the Association of Official Agricultural Chemists, held at Washington, D. C., November 6 to 9, 1896, is respectfully submitted for publication as Circular No. 2 of this Division. On account of the great cost of reprinting the official methods of analysis, it is recommended that the changes in and additions to official methods authorized by the association be printed separately for the guidance of analysts during the year 1897. They are to be used as complementary to the official methods published in Bulletin No. 46.

Respectfully,

H. W. WILEY,
Chief of Division.

Approved:

CHARLES W. DABNEY, Jr.,
Assistant Secretary.

CHANGES IN AND ADDITIONS TO METHODS OF ANALYSIS ADOPTED AT THE THIRTEENTH ANNUAL MEETING OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

[In all cases, unless otherwise mentioned, pages and paragraphs cited refer to Bulletin No. 46, Chemical Division of the United States Department of Agriculture.—H. W. W.]

METHODS FOR THE ANALYSIS OF FERTILIZERS.

DETERMINATION OF NITROGEN.

Page 21 (h), third line, substitute for "a large excess of magnesia," "5 grams or more of magnesium oxid free of carbon dioxid."

DETERMINATION OF POTASH.

Page 22 (2) (b), first and second lines changed to read: "When it is desired to determine the total amount of potash in organic substances like cotton-seed meal, tobacco stems," etc.

Page 24, (c) *Factors.*—The following are to be used for computing from the potassium-platinum chlorid:

(K_2PtCl_6) to KCl $\times$ 0.3069
to K_2SO_4 $\times$ 0.3587
to K_2O $\times$ 0.1939

DETERMINATION OF PHOSPHORIC ACID.

Page 14 (2) (*b*) (b_1), eighth line, omit "six," and insert "from ten to fifteen."

Sixteenth line, omit all after "wash" to end of sentence and insert "from five to six times with water, using in all from 150 to 250 cc."

METHODS FOR THE ANALYSIS OF SOILS.

Determination of Total Nitrogen.

Page 42, section 8, substitute the following: "From 7 to 14 grams of the soil are placed in a small Kjeldahl digesting flask, about 250 cc capacity, with 30 cc of strong sulphuric acid, or more, if necessary, and 0.7 gram yellow oxid of mercury, and boiled for an hour. The residue is oxidized with potassium permanganate in the usual way. After cooling, the flask is half filled with water, vigorously shaken, the heavy matters allowed to subside and the supernatant liquid poured into a flask of from 1,000 to 1,200 cc capacity. This operation is repeated until the ammonium sulphate is practically all removed and the large flask is a little more than half full. The distillation of the ammonia from the large flask is accomplished in the usual manner after the addition of the necessary alkali. If a sample is known to contain a considerable amount of nitrate, the modified Kjeldahl method, to include the estimation of nitrates, is to be substituted for the one above described."

METHODS FOR THE ANALYSIS OF FOODS AND FEEDING STUFFS.

The methods on page 62 (7) under (a), (b), and (c), are made official. The method on page 62 (d) is retained as a provisional method, with the following directions for preparing the reagent:

Fourth line from bottom take out "12 per cent."

Third line from bottom take out "in glacial acetic acid," and insert "acetate made by dissolving 12 grams of phenylhydrazin acetate and 7.5 grams of glacial acetic acid in sufficient water to make the volume 100 cc."

For the factors at the top of page 63 for calculating results, the following are substituted:

Weight of hydrazone $\times$ 0.516 + 0.0104 $\div$ weight of sample used = percentage furfurol.

Furfurol $\times$ 1.84 = pentosans.

Pentosans $\div$ 0.88 = pentoses.

Furfurol $\times$ 1.65 = xylan.

Furfurol $\times$ 2.03 = araban.

METHODS FOR THE ANALYSIS OF DAIRY PRODUCTS.

Changes in Methods of Milk Analysis.

Page 36 (c). Determination of Nitrogen Compounds.

Add at end of paragraph the following:

1. Provisional method for determination of casein in cow's milk.—The determination of casein in milk should be made when the milk is fresh, or nearly so. When it is not practicable to make this determination within twenty-four hours, add 1 part of mercuric chlorid to 2,000 parts of milk, and keep in a cool place. Weigh about 10 grams of milk, dilute in a beaker with about 90 cc of water at 40°–42° C., and add at once 1.5 cc of a solution containing 10 per cent of acetic acid by weight. Stir with a glass rod, and let stand from three to five minutes longer. Then decant on filter, wash two or three times with cold water by decantation, and then transfer precipitate completely to filter. Wash once or twice on filter. The filtrate should be clear, or very nearly so. If the filtrate is not clear when it first runs through, it can generally be made so by two or three repeated filtrations, after which the washing of the precipitate can be completed. The washed precipitate and filter paper are then digested as in the regular Kjeldahl method for the determination of nitrogen, and the process is completed as usual. To calculate the nitrogen into an equivalent amount of casein, multiply the per cent of nitrogen by 6.25.

In working with milk which has been kept with mercuric chlorid, the acetic acid should be added in small proportions, a few drops at a time, stirring after each

addition, and continuing the addition of acetic acid until the liquid above the precipitate becomes clear, or very nearly so.

2. Provisional method for determination of albumin in cow's milk.—The filtrate obtained above in separating casein is placed in a water bath and heated to the boiling temperature of water for ten or fifteen minutes. The filtered and washed precipitate is then treated by the Kjeldahl method for determining nitrogen. The amount of nitrogen multiplied by 6.25 gives the amount of albumin.

CHANGES IN METHODS OF CHEESE ANALYSIS.

Page 37, (a) *Preparation of sample.*—Substitute the following: "When the cheese can be cut, a narrow wedge-shaped segment reaching from the outer edge to the center of the cheese is taken. This is to be cut into strips and passed through a sausage-grinding machine three times, being mixed as completely as possible after each grinding, taking care to avoid evaporation of water. From the mass thus prepared samples are taken for analysis.

"When the cheese can not be cut, samples are taken by a cheese trier. If only one plug can be obtained, this should be taken perpendicular to the surface at a point one-third of the distance from the edge to the center of the cheese. The plug should reach either entirely through or only half way through the cheese. When possible, draw three plugs, one from the center, one from a point near the outer edge, and one from a point half way between the other two. For inspection purposes, the rind may be rejected; but for investigations requiring the absolute amount of fat in the cheese, the rind is included in the sample. It is preferable to grind the plugs in a sausage machine, but when this is not done, they are cut very fine and carefully mixed."

Page 37, (b) *Determination of water.*—Substitute the following for the first two lines: "From 2 to 5 grams of cheese should be placed in a weighed platinum or porcelain dish which contains a small quantity of material like freshly ignited asbestus, sand, etc." (Rest unchanged.)

Page 37, (c) *Determination of ether extract.*—Substitute the following: "Use a test tube containing a perforation in the bottom, or in place of this an extraction thimble of fat-free paper. When a test tube is used, place at the bottom some extracted cotton, or glass wool, or asbestus, and, in addition, wrap the lower end outside with filter paper to prevent particles of asbestus or copper sulphate being carried down through the perforation into the extraction flask. Pack the prepared tube as follows: Put first a mixture containing equal parts of anhydrous copper sulphate and pure incinerated sand, filling the tube for about 2 inches. Avoid packing too tightly. Upon this place a little asbestus or other suitable material. Next are added from 2 to 5 grams of the sample of cheese which has been weighed for analysis. It is convenient to place the weighed sample in a little cone of copper foil or of filter paper and slip this into the top of the tube containing the copper sulphate. Place the tube in a continuous extraction apparatus and exhaust with anhydrous ether. After five hours, the partially extracted cheese is removed and ground with pure sand in a mortar. This partially extracted cheese is brittle, and grinds up about as easily as a dry biscuit. After grinding with the sand, the cheese is replaced in the tube and the extraction continued ten hours longer."

Page 37, add: "(g) *Provisional method for the determination of acidity in cheese.*—Add water at a temperature of 40° to 10 grams of cheese until the volume equals 105 cc; agitate vigorously and filter. Titrate portions of 25 cc of filtrate, corresponding to 2.5 grams of cheese, with a standardized solution of sodium hydroxid, preferably one-tenth normal. Use phenolphtalein as indicator. Express amount of acid as lactic acid."

CHANGES FROM PROVISIONAL TO OFFICIAL METHODS.

Page 37, the methods described under (a), (b), (c), (d), (e), and (f) after modifying as described above.

CHANGES IN METHODS FOR THE ANALYSIS OF TANNING MATERIALS.

Page 77, 1. *Preparation of sample.*—Add at bottom of page: "Extracts must be heated to 50°, well shaken, and allowed to cool slowly to room temperature."

Page 78, 3. *Determination of moisture.*—First line, insert "5" instead of "2."

Page 78, 4. *Quantity of tanning material.*—Substitute the following: "In the case of barks, woods, leaves, etc., use such an amount of the material as to give about 1 gram of total solids per 100 cc of solution, and extract in a Soxhlet or similar apparatus. In the case of extracts, use 20 grams, dilute with water at 80°, cool slowly to 20°, and make volume up to a liter."

Page 78, 5. *Determination of total solids.*—First line, insert after "and," "without filtering."

First line, take out comma and insert period after "pipette," and add sentence, "Care must be taken to keep the solution at 20°."

Page 78, 6. *Determination of soluble solids.*—Substitute the following: "Filter about 125 cc of the solution measured, at 20°, through a double folded filter (S. and S. No. 590, 25 cm), returning the filtrate through the filter twice. Evaporate 100 cc as before."

Page 78, 7. *Determination of tanning substances.*—Substitute the following: "Prepare 20 grams of hide powder by shaking for five minutes with 250 cc of water and straining through linen. Repeat the operation three times. Finally, remove as much water as possible by squeezing in a press. Transfer the pressed hide powder to a covered dish, and weigh. Dry a portion approximately equal to one-fourth of the whole to constant weight at 100°. Add the remainder of the hide powder to 200 cc of the original unfiltered solution at 20°, and shake for ten minutes. Add 5 grams of barium sulphate free from soluble salts, and shake again for one minute. Filter immediately through a folded filter (S. and S. No. 590, 15 cm), returning the first 25 cc. Evaporate 100 cc as before. The weight of the residue must be corrected for the dilution caused by the water contained in the hide powder. The shaking must be done in some form of mechanical shaker. The simple machine used by druggists, and known as the 'milk-shake,' is recommended."

Page 78, 2. *Testing the hide powder.*—Substitute the following:

"(a) Shake 10 grams of the hide powder with 200 cc of water for five minutes; filter through muslin or linen; squeeze out thoroughly by hand; collect the hide powder, and repeat the operation twice with the same quantity of water. Pass the last filtrate through paper until a perfectly clear liquid is obtained. Evaporate 100 cc of the final filtrate in a weighed dish; dry at 100° until the weight is constant. If the residue amount to more than 10 mg, the sample should be rejected. The hide powder must be kept in a dry place and tested once a month.

"(b) Prepare a solution of pure gallotannic acid by dissolving 5 grams in 1 liter of water. Determine the total solids by evaporating 100 cc of this solution and drying to constant weight. Treat 200 cc of the solution with hide powder exactly as described in paragraph 6. The hide powder must absorb at least 95 per cent of the total solids present. The gallotannic acid used must be completely soluble in water, alcohol, acetone, and acetic ether, and should contain not more than 1 per cent of substances not removed by digesting with excess of yellow mercuric oxid on the steam bath for two hours."

Page 78, add the following:

"8. Testing the Nontannin Filtrate.

"(a) *For tannin.*—Test a small portion of the clear nontannin filtrate with a few drops of a 10 per cent solution of gelatin (Nelson's). A cloudiness indicates the presence of tannin, in which case the determination must be repeated, using 25 grams of hide powder instead of 20 grams.

"(b) *For soluble hide.*—To a small portion of the clear nontannin filtrate add a few drops of the original solution, previously filtered to remove reds. A cloudiness indicates the presence of soluble hide due to incomplete washing of the hide powder. In this case, repeat the determination with perfectly washed hide powder."

METHODS FOR THE ANALYSIS OF FERMENTED AND DISTILLED LIQUORS.

Add on page 77, at end of Section VII:

"19. Determination of Fusel Oil.

"The apparatus recommended for this determination is Bromwell's modification of Roese's fusel-oil apparatus.

"This apparatus consists of a pear-shaped bulb holding about 200 cc, stoppered at the upper end and sealed at the lower to a graduated stem about 4 mm in internal diameter. To the lower end of this graduated stem is sealed a bulb of 20 cc capacity, the lower end of which bears a stopcock tube. The apparatus is graduated to 0.02 cc, from 20 cc to 22.5 cc.

"The reagents required are fusel-free alcohol that has been prepared by fractional distillation over caustic soda or caustic potash, and diluted to exactly 30 per cent by volume (sp. gr.,0. 96541), chloroform freed from water and redistilled, and sulphuric acid (sp. gr. 1.2857 at 15.6°).

"Distill slowly 200 cc of the sample under examination till about 175 cc have passed over, allow the distilling flask to cool, add 25 cc of water, and distill again till the total distillate measures 200 cc. Dilute the distillate to exactly 30 per cent by volume (sp. gr., 0. 96541 at 15.6°).

"The following is an accurate method for diluting any given alcohol solution to a weaker solution of definite percentage: Designate the volume percentage of the stronger alcohol by V, and that of the weaker alcohol by v. Mix v volumes of the stronger alcohol with water to make V volumes of the product. Allow the mixture to stand till full contraction has taken place, and till it has reached the temperature of the original alcohol and water, and make up any deficiency in the V volumes with water.

"*Example.*—It is desired to dilute a distillate containing 50 per cent of alcohol by volume until it contains 30 per cent. To 30 volumes of the 50 per cent alcohol add enough water to make 50 volumes, or place 150 cc of the distillate in a 250 cc flask, fill to the mark with water, mix, cool, and fill to the mark again.

"Prepare a water bath, the contents of which are kept at exactly 15°, and place in it the apparatus (covering the end of the tube with a rubber cap to prevent wetting the inside of the tube), and flasks containing the 30 per cent fusel-free alcohol, chloroform, sulphuric acid, and the distillate diluted to 30 per cent by volume. When the solutions have all attained the temperature of 15°, fill the apparatus to the 20 cc mark with the chloroform, drawing it through the lower tube by means of suction, add 100 cc of the 30 per cent fusel-free alcohol and 1 cc of the sulphuric acid, invert the apparatus, and shake vigorously for two or three minutes, interrupting once or twice to open the stopcock for the purpose of equalizing pressure. Allow the apparatus to stand ten or fifteen minutes in water that is kept at the temperature of 15°, turning occasionally to hasten the separation of the reagents, and note the volume of the chloroform. After thoroughly cleansing and drying the apparatus, repeat this operation, using the diluted distillate from the sample under examination in place of the fusel-free alcohol. The increase in the chloroform volume with the sample under examination over that with the fusel-free alcohol is due to fusel oil, and this difference (expressed in cubic centimeters), multiplied by the factor 0.663, gives the volume of fusel oil in 100 cc, which is equal to the percentage of fusel oil by volume in the 30 per cent distillate. This must be calculated to the percentage of fusel oil by volume in the original liquor.

"*Example.*—A sample of liquor contains 50 per cent of alcohol by volume. The increase in the chloroform volume with the 30 per cent fusel-free alcohol is 1.42 cc. The increase in the chloroform volume with the distillate from the liquor under examination, diluted to 30 per cent, is 1.62 cc. Difference, 0.20 cc. The volume of fusel oil in 100 cc of the 30 per cent distillate then is $0.20 \times 0.663 = 0.1326$ cc, and by the proportion $30 : 50 :: 0.1326 : 0.221$, we obtain the percentage of fusel oil by volume in the original liquor.

"20. Determination of Aldehydes.

"(a) *Preparation of reagent.*—Eighty cubic centimeters of a saturated solution of sodium disulphite are mixed with a solution of 0.12 gram of fuchsin in about 800 cc of water, 12 cc of sulphuric acid added, the solution thoroughly mixed, and diluted with water to 1 liter.

"(b) *Determination.*—A portion of the sample is diluted with water, or strengthened with aldehyde-free alcohol until it contains 50 per cent of alcohol by volume, and 25 cc of this solution are treated with 10 cc of the reagent, and allowed to stand twenty minutes. At the same time 25 cc of a solution of 0.05 gram of acetic aldehyde in 1,000 cc of 50 per cent alcohol are treated in the same manner and allowed to stand the same length of time. The relative intensity of the colors of the two solutions is then determined by means of a colorimeter, and from the figure thus obtained the weight of aldehyde is estimated as acetic aldehyde, and calculated to percentage of the original liquor.

"21. Determination of Ethereal Salts.

"After the determination of the volatile acids, the neutralized distillate is transferred to a flask connected with a reflux condenser, treated with 25 cc of tenth normal sodium hydroxid, and boiled one-half hour. The flask and contents are then cooled, 25 cc of tenth normal hydrochloric acid added, and the excess of acid titrated with sodium hydroxid, using phenolphtalein as indicator. The number of cubic centimeters of tenth normal alkali used in this titration, multiplied by 0.0088, is equal to the weight in grams of ethereal salts (calculated as ethyl acetate) in the volume of liquor taken for the determination."

OPTIONAL METHOD OF DETERMINING PHOSPHORIC ACID IN SOILS.

Note.—The following method, devised by Mr. Goss, was recommended as an optional method by the association in 1895, but was not ordered printed with the official methods. A synopsis of it is printed here for the convenience of analysts:

"Ten grams of the air-dried soil, passed through a sieve of one millimeter mesh, are placed in a small Kjeldahl flask marked at 250 cc. From 20 to 30 cc concentrated sulphuric acid and approximately 0.7 gram yellow oxid of mercury are added, the contents of the flask well mixed by shaking, and oxidized over the open flame, as in the determination of nitrogen, for an hour. After cooling, about 100 cc of water, 5 cc of concentrated hydrochloric acid and 2 cc of concentrated nitric acid are added, and the mixture reboiled to oxidize the iron, cooled, and the volume completed to one-fourth of a liter with water, and the contents of the flask filtered through a dry, folded filter paper. One hundred cubic centimeters of the filtrate are placed in a flask of about 450 cc capacity, strong ammonia added until a permanent precipitate is formed, which is dissolved by the addition of about 7 cc of nitric acid, and the mixture boiled until clear. The flask is removed from the flame and cooled at room temperature for exactly two minutes, 75 cc molybdate solution added, and the flask placed in a water bath kept at 80° for 15 minutes, shaking vigorously four or five times meanwhile. After removing from the bath, the flask is allowed to stand for ten minutes until the precipitate has settled, and the supernatant liquid is poured on to the filter paper under pressure, the precipitate being partially brought upon the paper. The flask and precipitate are thoroughly washed with ammonium-nitrate solution, the precipitate either by decantation or in the filter paper. The flask is then placed under the filter, the precipitate is dissolved in ammonia, and the phosphoric acid estimated by the usual processes. Details of the manipulation are given in Bulletin No. 43 of the Division of Chemistry, pp. 58–60."

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